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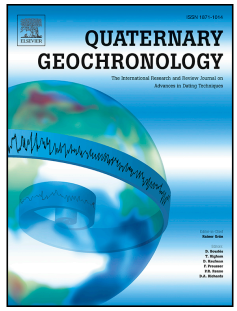

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AMS dating of insect chitin - A discussion of new dates, problems and potential

Eva Panagiotakopulu[1*], Thomas F. G. Higham[2], Paul C. Buckland[3], Jennifer A. Tripp[4], Robert E. M. Hedges[2]

[1]School of GeoSciences, University of Edinburgh, Drummond Street, EH8 9XP, UK

[2]Research Laboratory for Archaeology and the History of Art, Dyson Perrins Building, South Parks Rd, Oxford, OX1 3QY, UK

[3]20 Den Bank Close, Sheffield, S10 5PA, UK

[4] Department of Chemistry & Biochemistry, San Francisco State University, 1600 Holloway Avenue, San Francisco, California 94132, USA

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Corresponding author:

E. Panagiotakopulu

E-mail: eva.p@ed.ac.uk

Abstract

Results from AMS dating applied to insect chitin from a variety of contexts and different preservation conditions and retrieval methods are presented. Secure contexts, which include other dated organic material from different geographic locations ranging from Egypt to Greenland and different chronological periods, from Lateglacial to Medieval, have been used. In addition, insect species with different dietary requirements have been selected for dating purposes in order to provide an understanding as to whether diet plays a role in the chitin dating results. Dates from each context/site are discussed separately in the context of their stratigraphy and/or archaeology. Our research concentrates on the results from pre-treatment methods which require small quantities of chitin as these could be applied in a variety of Quaternary and archaeological contexts. The dates from carbonised and desiccated remains where no chemicals had been involved in storage fell within the range of dates from other organics or the archaeology. Although some of the dates from waterlogged contexts were successful, problems were encountered and these have been linked with long term storage in various alcohols of uncertain provenance. Whilst short term immersion in paraffin (kerosene) and alcohols during processing probably has no impact, it is recommended that chitinous material for dating be stored in acidified distilled water. Our results demonstrate the potential of chitin as a dating medium and provide a basis for its wider application.

1. Introduction

The remains of the chitinous exoskeleta of insects are one of the most common identifiable remains in archaeological and Quaternary sediments. As well as providing an important tool in reconstructing past environments (Elias, 2010), they are also potentially a source of carbon for ^{14}C dating by accelerator mass spectrometry (AMS). Until the development of accelerator dating, recovery of a sufficient mass of closely stratified insect remains for a date was usually considered impractical. Even where remains were frequent – and the similar case of dating the limits of the last glaciation in the English Midlands by picking out 34000 opercula of the snail *Bithynia tentaculata* from deposits at Trysull in Staffordshire is instructive (Shotton in

Morgan, 1973) – the time investment was often counter-productive and either bulk dates on undifferentiated organic materials or on plant macrofossils were preferred. All this changed with the development of AMS dating and with it the ability to date increasingly small amounts of material. Single charred seeds were dated (e.g. Jones and Legge, 1987) but attempts to date insects often produced results at variance with those obtained on other materials from the same context; the dates from chitin were older. In some cases, these could be explained in terms of hard water effect in either bulk sediment or moss dates, but paired dates on insects and terrestrial plant macrofossils occasionally produced dates several thousand years apart (e.g. Snyder et al., 1994). Elias and Toolin (1990) obtained disparate dates on insects from the same context, interpreting this as a mixed assemblage and raising issues associated with taphonomy as an explanation. Certainly it seemed probable that aquatic taxa, such as larval chironomids, might inherit some hard water effect from either prey or water body (e.g. Fallu et al., 2004). This seemed less likely with purely terrestrial species, yet both predatory and graminivorous ground beetles had produced apparently aberrant dates.

Some of the problems reflected pre-treatment and these have largely been solved by improvements in the sample purification technique (e.g. Hodgins et al., 2001; Tripp et al., 2004; Tripp and Higham, 2011). In this paper we present research undertaken using the pre-treatment methodology of Tripp et al. (2004) (see also Tripp and Higham, 2011). We discuss selection of the samples and results in a move towards a standardised methodology for dating insect chitin.

2. Materials and Methods

2.1 Dating methodology

Tripp et al. (2004) described two methods for pretreating insects prior to AMS dating. The method applied in this paper is termed method IA*, designed for small or fragile remains. We took identifiable insect remains and treated them with a solvent wash (acetone, methylene chloride, and acetone again) in a 12ml glass test tube. Following this the material was freeze-dried for ~five hours. The remains were then added to a 0.5 M HCl solution for three days at room temperature (RT), ezee-filtered™ and rinsed with ultrapure water, then freeze-dried again.

The Oxford Radiocarbon Accelerator Unit (ORAU) also has a method designed for higher weight or well-preserved insect material (denoted IB*) that isolates chitosan, the deacetylated chitin. In this method 5 mL 50% NaOH is added to the test tube following the acid wash for 30 min at 70°C (mainly chitin is left after this procedure). The solution is ezee-filtered™, retaining solids, then rinsed with ultrapure water. After this small volumes of 6 M HCl are added to make the solution weakly acidic (pH=3) and thereby dissolve the chitin. The solution is then filtered, discarding the insoluble fraction. The chitin is reprecipitated by making the solution strongly acidic through adding 6M HCl. The chitin is recovered using pre-combusted glass fibre papers to retain the solids, which are then freeze-dried prior to combustion, graphitisation and AMS dating. In this paper, we used Method IA* for all dated samples because the material was small and fragile, and using Method IB* would have resulted in the complete loss of all sample material. Reference to radiocarbon dates in the paper follow the conventions outlined by Millard (2014).

2.2 Palaeoecology

We have further tested the reliability of the methodological approach outlined by Tripp et al. (2004) by dating more materials from different preservational contexts, from desiccated through anaerobic to permafrost, and with different post-recovery histories, from dry storage in museums to storage in alcohols. Samples chosen for dating represent a variety of sites which range from Egypt to Greenland (Figure 1) with diverse preservation and a variety of species in order to address the breadth of the palaeoecological assemblages. The contexts chosen were either closely dated by independent means (e.g. based on their archaeology) or, in the case of the Lateglacial site, allowed us to compare the AMS dates from chitin with other dates from other dated proxies as well as other dating information (Table 1). Several of the archaeological case studies chosen have proven controversial in terms of chronology (rewrapping of Mummy 1770, Santorini eruption, the end of Norse Greenland). We hoped that new determinations of chitin might help to resolve certain of these ongoing problems. In the case of the Lateglacial site, dated plant material and qualitative research provided a dating framework (see discussion below).

The parameters set for the dating methodology were developed during the time of the project and various factors which could have affected the dates were examined. Insect diets were taken into account in order to understand whether this affected dating results (Table 2). The charred insects were retrieved through dry sieving over a 300 μm sieve (see Panagiotakopulu, 2000). No chemicals were involved during sorting and storage. The desiccated insects were retrieved during examination of the mummies, and no further processing was involved. Material from waterlogged deposits were recovered using the standard technique for insect recovery, paraffin (kerosene) flotation, sorting of the residue and storage in 70% ethanol (Coope and Osborne, 1968). The tubes were thereafter topped up with alcohol periodically. No records were kept as to whether ethanol or methanol was used in storage. In any case, attempts to find out whether the alcohols and paraffins were of wood or oil origin with the suppliers were unsuccessful. In some cases (discussed in the text) the insects were mounted on cards using the natural sap glue gum tragacanth. Additional samples from waterlogged sediments from Greenland were sorted in water, without any chemicals involved, and material was stored in acidified water. Although this was a lengthy process, the quantity of the material produced for dating was insufficient and this process was abandoned; similar problems were met with in material from the Last Glacial Maximum site at Dimlington in East Yorkshire (Bateman et al., 2011). As a realistic alternative for reducing the use of chemicals, processing using the standard methodology and storage of the material recovered in acidified water has been adopted.

3. Results

3.1 Lateglacial

3.1.1 Samples

One approach to the validation of the use of insect chitin for AMS dating is by parallel dates on plant and insect material. As part of the preparation of the Quaternary Research Association field guide to East Yorkshire and North Lincolnshire (Bateman et al., 2001), it was decided to obtain additional dates on an insect fauna recovered from a compressed peat horizon exposed during road construction in 1976 at Armthorpe, South Yorkshire (NGR SE 637058). A bulk sediment date on adjacent site had been obtained previously by Gaunt et al. (1971) (Table 3), but the relatively thermophilous nature of the insect fauna, with an MCR predicted summer range of 15-16°C and winter of -12 to + 1°C (Buckland, 2001 and unpubl.), suggests that the date should be about a thousand years older, during the warmer part of the Lateglacial Interstadial. The species selected for dating was the reed beetle *Plateumaris sericea* (L.), a phytophage found on a range of aquatic plants (Cox, 2007), and recorded by Stainforth (1944) as breeding on *Typha latifolia* L., *Iris pseudacorus* L., *Scirpus lacustris* L., *S. maritimus* L. & *Sparganium ramosum* (Curt.).

3.1.2 Discussion

Although there are continued reservations over the AMS dates (Table 3), based on the entomology, these are close to Gaunt's original bulk peat date (Gaunt et al., 1971) and plant macrofossil and insect dates show virtually complete overlap. A similar problem with dates evidently too young was noted by Buckland (1984) in relation to Lateglacial organic material from beneath blown sands at Messingham in North Lincolnshire and dating by OSL (Bateman, 1995) has confirmed the offset in the radiocarbon dates, although currently no explanation is offered for the discrepancy.

3.2 Late Bronze Age Aegean

3.2.1 Samples

Akrotiri is a settlement site on the island of Santorini in the Aegean which was destroyed by a Plinian volcanic eruption during the late Bronze Age (Doumas, 1992). As a result of the significance of the date for the chronology of the eastern Mediterranean, there has been a long discussion about the dating of the eruption which is thought to have played an important role in the region. The generally accepted dates of 1627 cal BC-1600 cal BC, from an olive branch found adjacent to the site (Friedrich et al., 2006, Heinemeier et al., 2009) have recently been questioned by Cherubini and colleagues (2014) (see also Manning, 2014; Wiener & Earle, 2014). The organic material found inside the settlement of Akrotiri, including material from the pithoi of the West House, was charred as a result of the pyroclastic flow (see Druitt, 2014) that destroyed the site. Charred stored pulses mainly *Lathyrus clymenum* L. from the storerooms of the West House were infested with bruchids, *Bruchus rufipes* L., and these have been dated in order to compare results with existing dates from plant remains and to obtain additional dating information from a context inside the settlement during its final phase (see also Panagiotakopulu et al., 2013).

3.2.2 Discussion

Dates from botanical remains have been obtained from plant remains from several pithoi within the West House (Housley et al., 1990). Although the aim of the dating programme was to provide a chronology for the eruption, the results ranged quite broadly and did not provide a narrow date (for recent discussion, see Manning, 2014). For this study the comparison has been restricted to material from the same pithos from room 5 on the ground floor of the West House, pithos 1, to constrain the methodology as closely as possible (Table 4). The remains of *Bruchus rufipes* from pithos 1 provided a date of 3368 ±29 BP which ranges between 1744 cal BC and 1538 cal BC (at 95.4% probability) and fits closely with estimates for the age of the eruption based on other data (e.g. Manning et al., 2006).

Although it does not provide any further refinement to the dates already obtained by the sequence of dates from an olive tree branch (Friedrich et al., 2006) or Bayesian modelled archaeological sites (Manning et al., 2006), the date agrees with that provided from the seeds in the same sealed context.

3.3 Pharaonic and Roman Egypt

3.3.1 Manchester Mummy 1770

The Manchester Museum has an extensive collection of Egyptian materials, including several mummies with evident insect infestation. Whilst some of this reflects post-excavation attack – museum beetle, *Anthrenus museorum* (L.) is a common contaminant -, others clearly relate to the decay of the body. Mummy 1770, probably from Hawara in Middle Egypt, has been the subject of detailed investigation as part of the Manchester Mummy Project (David, 1979), and insect remains were recovered during the unwrapping process. The calliphorid fly *Chrysomya albiceps* (Weide.) was found between the wrappings of the mummy (Curry, 1979), and as the species is not associated with dried flesh (Smith, 1986), it had clearly entered the bandages during the wrapping process. At the present day it is widespread in Egypt, feeding on carrion (Omar 1995), and its maggots, along with those of the cheese skipper *Piophila casei* (L.), also recovered from the mummy (Curry, 1979), probably provided the prey for the small clerid beetle *Necrobia rufipes* (Deg.). The last is a rare casual introduction to Britain, since it requires a minimum temperature of 22°C to establish breeding populations (Haines and Rees, 1989). It must have been a widespread accompanist to the embalmers, although it appears to prefer dried meat (Koch, 1989). The human body, that of a female of 13-14 years, showing parasitism by the guinea worm, *Dracunculus medinensis* (L.), had suffered considerable decay before mummification. The lower legs and the feet were missing and replaced with prosthetics (Isherwood et al., 1979). In addition, the discrepancy in the radiocarbon dates produced during the Manchester Mummy project, with the bones providing dates of 1426 cal BC - 510 cal BC (right scapula) and 1493 cal BC - 546 cal BC (left scapula) and the bandages dating to several hundred years later (cal AD 140 - cal AD 659 (outer bandage) and cal AD 25 - cal AD 605 (part 4 bandage)) (see Table 5), led to the suggestion that the body had been re-wrapped several centuries after its primary interment (David, 1978). This would not have been an unusual occurrence where royal mummies are concerned, the remains having been recovered from robbed tombs and re-entombed (cf. Buckland and Panagiotakopulu, 2001). Further dates from mummy 1770 (BM-1602, 407 cal BC - 41 cal BC (left humerus) and BM-1839, 161 cal BC - cal AD 418 (linen)) (Burleigh et al., 1982) were subsequently corrected by the British Museum Laboratory and the second one rejected (Bowman et al., 1990). The date from the left humerus of the mummy (BM-1602) was revised to 511 cal BC - cal AD 259. A new set of date from La Jolla (Linick 1984) contributed further to the debate without providing resolution for the discrepancies. The dates obtained from skin tissue from the left humerus (LJ-4915, from 408 cal BC - 208 cal BC) and dates from the linen bandages (LJ-4995, arm and chest bandages over the right side, 1282 cal BC - 932 cal BC and LJ-4996, bandages beneath the cartonnage mask, 362 cal BC - 3 cal BC). A new set of dates from linen bandages has added to the existing information (Cockitt et al., 2014). The linen sampled from beneath the cartonnage mask provided a date, OxA-11650, of 133 cal BC - 323 cal AD, diverging from the La Jolla cartonnage date, while linen from the 16th layer from the top over the legs was dated (OxA-11650) from 358 cal BC to 58 cal BC. Two of the dates (LJ-4996, OxA-11650) are very similar, indicating that the mummy could be Ptolemaic (Cockitt et al. 2014). Although these authors proposed that discrepancies among the different dates from linen bandages could be the result of "repairs" during the Roman period, the issues with the dates are probably a result of the substances used during the mummification process. Discordant dates on an ibis and its linen wrappings (Gove et al., 1997) have highlighted the problems caused by the use of bitumen and other natural substances during the mummification process. Recent research, developed from dating asphalt impregnated bone remains from Rancho La Brea, indicates the potential of the technique (Fuller et al., 2014) and a similar approach should be developed for bitumen covered materials from Egyptian mummies.

3.3.2 Discussion

The new date from the insects sampled from underneath the upper right leg of Mummy 1770, OxA-2517, 352 cal BC - 62 cal BC (Table 5) is similar to the later date from the linen beneath the cartonnage mask, LJ-4996 (Linick 1984) and is virtually the same date as the new date from linen directly above the legs, OxA-11650 (Cockitt at al., 2014), indicating that 1770 is Ptolemaic, as opposed to New Kingdom or Roman. If the first set of dates on the bones is discounted, the necessity of arguing for a re-wrapping of the corpse several hundred years after its initial mummification ceases to be a problem. The new date on the insect remains firmly places the mummy during the Ptolemaic period. The older dates can be explained by the use of bitumen during mummification but there is no need to invoke contamination with beeswax and leaf gelatin in the outer bandages (see David, 1979; 2000) or repairs during the Roman period to explain the apparently slightly younger dates from bone and bandage. Although Hodge and Newton (1979) regard the possibility of contamination with bitumen (see David, 1978) as unlikely because of careful pre-treatment, dating of insects embedded in bitumen from a Greco-Roman Turin funerary facemask (discussed below) indicates that it is possible for bitumen to have penetrated deeply into the materials being dated.

3.3.3 Turin

The Collections of the Turin Museum include several cartonnage face masks, which would have been put over the face of the dead during burial, and one (Mus. Suppl. No. 14271), from Assiut, preserves several complete individuals of the dermestid beetle *Dermestes frischii* Kugelann attached in a black resinous substance to the back of the mask (Panagiotakopulu, 2003). The species is widespread around the Mediterranean (Ferrer et al., 2004), but is a rare import to Britain at the present day (Peacock, 1993). It has been recorded previously in New Kingdom deposits at the Workmen's Village at Amarna in Middle Egypt (Panagiotakopulu, 1999), in Roman mummies in the Dakhleh Oasis, where it is associated also with *D. leechi* Kalik and *N. rufipes* (Lord, 2011), and in the mummy of an ox, now in the Munich Museum collection (Seifert, 1987). Dermestids would have been a frequent problem for those involved in embalming bodies (cf. Strong, 1981), and there can be no doubt that the beetles were contemporary with the body. The mask can be dated stylistically to the Greco-Roman (Ptolemaic-Roman) period, ca. 323 cal BC – cal AD 325, part of a tradition that goes back to the Early Dynastic period (Riggs, 2002).

3.3.4 Discussion

Despite the careful cleaning of the complete insect specimens and the pre-treatment, it is apparent that the date is affected by contamination, most probably a large part of the date, OxA-X-2347-9, 8791 cal BC - 8606 cal BC (Table 5) has been contributed by contaminant material, most probably the material described as 'resinous' in the Museum. Resins, however, would have come from contemporary trees and would not have a significant impact on the date and it seems probable that the source of contamination is bitumen, widely used in mummification in the Greco-Roman period (Serpico, 2000).

3.4 Roman Britain

3.4. 1 Empingham, Rutland

For much of the Roman period, historical context and coins provide a reasonably secure chronology, which can be extended to the often abundant finds of pottery. This gives a suitable framework for validating other dating techniques. The construction of Rutland Water, the largest man-made lake in western Europe, in the valley of the river Gwash, 30 km east of Leicester, in 1967-73 was accompanied by a series of under-resourced rescue excavations directed initially by the late Malcolm Dean and later by Sam Gorin. Of the eleven sites excavated, Site 1 included a stone-lined well, 0.7 m in diameter and 5 m deep, set in a cobbled area adjacent to a stone-footed rectangular building, 21.4 m by 10.5 m (Cooper, 2000). Ceramic evidence indicates that the well had been filled in during the last quarter of the third century AD. In the absence of support for palaeoecological research, Bob Alvey, then the technician in the University Museum at Nottingham, was able to take a single bulk sediment sample from the filling of the well to process for plant macrofossils and insects, although as sorting was done by eye without a microscope, only the larger individuals of Coleoptera were recovered and passed to one of us (PCB) for report (Buckland, 1986; 2000). It is uncertain how the material was stored over the decade after its excavation but when passed to Buckland, the material was dry and in a glass tube. Subsequently, identifiable insects were mounted onto card with gum tragacanth and the remaining material returned to the tube. It is the latter material that was used to provide the date, which was obtained on the unidentified legs of large Carabid beetles.

3.4.2 Discussion

The calibrated date range of 50 cal BC – cal AD 70 (OxA-19603, Table 6) is clearly too early by at least two hundred years. In the absence of evidence for pre-Roman settlement on the site, it is difficult to argue for a local source of contamination. In any case, the range of ventral sclerites femora and tibiae of Carabids, a large family whose members have diverse diets, used for the date renders this highly improbable. Identified taxa include both terrestrial predatory and graminivorous species but the aberrant date cannot be explained in terms of dietary preferences of particular species. The date remains enigmatic, although, with hindsight, it seems probable that at some stage during storage the glass tube was topped up with an alcohol derived from a fossil hydrocarbon source.

3.4.3 Lynch Farm, Peterborough

The problems with the Empingham material suggested that a more directed approach was necessary and a series of samples, of material identified to the species level, from a sample whose post-excavation history was better known was clearly required. The Roman settlement at Lynch Farm, Orton Longueville, on the Nene floodplain, near Peterborough, was excavated by Adrian Challands, Geoff Dannell and J.-P Wild for the Nene Valley Research Committee in 1972 in advance of gravel extraction. Interim reports were included in the annual summaries of results in *Durobrivae* and *Northamptonshire Archaeology*, and the final report on the site is currently in preparation (Upex, pers. comm.). Vicki Hughes (1995) examined an insect fauna from a well on the site as part of a Sheffield University MSc and an edited version of this work will appear in the final volume. The well, about 80 cm square and stone-lined, was less than two metres deep and had been filled in during the Roman period. The archaeological dates are based on evidence from coins from the backfilling of the well, in particular a coin of Theodora or Helena (cal AD 337 - 341) and three others with a date range of cal AD 206 - 402 (Walton in Upex forthcoming) and this places the structure in the mid fourth century or later. The sample for the insects, provided by J.-P Wild, consisted of approximately 2.5 kg of silty sediment with evident insect remains. Dates were obtained on three species of larger Carabid, *Nebria brevicollis* (F.), *Pterostichus niger* (Schall.) and *Amara aulica.* The first appears to feed mainly on maggots (Luff, 1998), Collembola and mites (Crowson, 1981), *P. niger* on insect larvae (Lindroth, 1986) and the last is largely graminivorous, feeding on the seeds of Asteraceae (*idem*.).

3.4.4 Discussion

The range of dates on insects from the same context at Lynch Farm (OxA-19574, OxA-19602, OxA-19572, OxA-19573, OxA-10599) (Table 6) largely agree with the archaeological dates; three, at 95% probability, include the date predicated by the archaeological evidence, although the date from *P. niger* which was mounted using the organic adhesive gum tragacanth is slightly younger than the two dates from *N. brevicollis* specimens stored in glass tubes in ethanol. The one date on a graminivorous beetle, on *Amara aulica* (Panz.), also mounted on cards using gum tragacanth, is also significantly younger than the others. It is possible that the tragacanth, derived from the sap of Middle Eastern species of the genus *Astragalus*, is responsible for erroneous results, but it is difficult to be certain that the reasons lie in biochemistry rather than taphonomy.

The overlap of four of the five dates is sufficient to suggest that the well was finally infilled towards the end of the Roman period, perhaps into the fifth century AD. Kenward (1976 and in Hall et al., 1980) has pointed out that the open nature of many well fills can allow ingress by either species which are largely subterranean or by individuals seeking hibernation or aestivation sites. It is possible that the post-Roman dates on *A. aulica* reflect the latter, although they would need to coincide with years of lower water table, when the floodplain was significantly drier than in the late Roman period, since this species is unlikely to seek out wetlands.

3.5 Norse Greenland

3.5.1 Gården under Sandet

The Norse settlement and abandonment of south-west Greenland has a reasonable historical record, from saga sources with their foundation myth of Erik the Red beginning settlement in the late tenth century to the final documentary reference to the more southerly Eastern Settlement in 1408 (Jansen, 1972). Radiocarbon dates largely support this evidence, with the abandonment of the more northerly Western Settlement sometime in the mid-fourteenth century, when the Bishop’s reeve Ivar Barðarson, is alleged to have visited and found only domestic animals (Panagiotakopulu et al., 2007). Excavation of the site at Gården under Sandet (GUS), south-east of the head of Ameralik fjord in the former Western Settlement, in the early 1990s (Berglund 1998) provided material for a range of palaeoecological studies (e.g. Buckland et al., 1998; Hebsgaard et al., 2009; Panagiotakopulu et al., 2007; Ross, 1997; 2004; Ross and Zutter, 2007; Schweger, 1998) and further research is ongoing.

3.5.2 Discussion

A series of samples was taken to compare results of AMS dating from different materials, including insects (Table 7). The first pair of dates examined charred seaweed and numerous puparia of the carrion fly *Heleomyza borealis* Bohe., found together in a soapstone container. The assemblage was interpreted as the residue of meat storage in seaweed ash (Buckland et al., 1998), something for which there is ethnographic evidence in the Outer Hebrides (Martin, 1695). Martin (*op. cit.*) refers specifically to preservation of seal meat with seaweed ash. The changing diet of the Norse Greenlanders has been examined in terms of isotopic composition of human bones (Arneborg et al., 1999; Nelson et al., 2012) and this shows a significant marine component (Arneborg et al., 2012). The GUS pot contained no bones, and it seemed possible that any deviation in date on the fly puparial exoskeletons, the maggots having fed on the pot contents, could be a reflection of marine reservoir effect and thereby contain a trace after seal meat. The charred seaweed, bladder floats of *Fucus vesiculosus*, provided a date (OxA-10531) (Table 7), which, as expected, was significantly skewed by the reservoir effect, and its $\delta^{13}C$ is also markedly enriched. If the reservoir effect is accepted as ~500 years (Reimer and Reimer, 2013, McNeely et al., 2006, Olsson, 1980), although there is some doubt, both spatially and temporally about this (cf. Ascough et al., 2006), then both the date on seaweed and that on the fly puparia should fall within the early part of the occupation of the farm, in the eleventh to twelfth century. It seems probable that the maggots were feeding on seal meat in the pot, something which the δ^{13}C figure would also support.

The range of dates from context 2790 is more problematic because while a twig of birch or willow provides a similar date to that from a sheep or goat dung pellet, placing both firmly in the first half of the Norse period, the insect dates are markedly discordant. The δ^{13}C ratio of the herbivore dung pellet is sufficiently close to indicate a terrestrial plant diet for the source,, something which pollen analysis of a similar pellet, producing 80% *Betula* pollen (Craigie, pers. comm.) would also imply. Two species of insect, a small rove beetle *Xylodromus concinnus* (Marsh.), and the latridiid *Latridius pseudominutus* Strand, selected because they are accidental Norse imports to Greenland (Buckland and Panagiotakopulu, 2010, Panagiotakopulu, 2014), have similar δ^{13}C ratios to the wood and dung. The puparia of *H. borealis* provide a ratio closer to that of the specimens associated with the seaweed. This may be a reflection of their respective feeding habits. *L. pseudominutus* feeds on moulds, spores and fungal hyphae (Böcher, 1988) and *X. concinnus* is probably carnivorous (Hinton, 1945) in similar habitats, mouldy hay and related materials in farms and outhouses in Norse Greenland, whilst maggots of the fly *H. borealis* feed on carrion, although occasional occurrences in plant materials suggest that heleomyzids can also be predatory (cf. Smith, 1986). It is possible that the food source of the last included significant amounts of marine material, presumably seal meat, but this cannot explain the discrepancies in dates, either between the puparia and the beetles or the three insect dates and those on other materials. The primary difference is in storage over the few years between processing and dating; the wood and dung pellet were stored dry in glass tubes, the insects were stored in 70% ethanol, again in all likelihood derived from fossil hydrocarbons.

In the light of this, a new sample was selected. Although processed by paraffin flotation, and sorted in ethanol, storage was either dry or in acidified distilled water. The sample (2469) comes from sediment accumulating in a pool formed in a hollow in the collapsed roof of the farm and must date from shortly after the farm's abandonment, since the presence of sheep ectoparasites clearly indicates that domestic animals were still returning to the site to drink (Panagiotakopulu et al., 2007). Two species were dated, puparia of a fly, *Scatella* cf. *stagnalis* (Fallén), which feeds on algae by eutropic pools (Ólafsson, 1991), and the true bug, *Nysius ericae groenlandicus* Zett., noted as feeding on a wide range of seeds (Böcher, 1972). The results, OxA-19576, cal AD 1255 - cal AD 1381 and OxA-19757, cal AD 1227- cal AD

1380 (Table 7), are consistent with the probable abandonment date for the farms in the Western Settlement in the middle of the fourteenth century (Panagiotakopulu et al., 2007).

Whilst these dates are useful for positioning the final abandonment, attempts to date the primary occupation with samples (3159) from the floor of the long house, sealed beneath the later centralised farm (Albrethsen and Ólafsson, 1998) were less successful. The two samples were sorted in water and the recovered insect remains preserved in distilled water. Without floating, this was a slow and inaccurate process with little recovered. A composite sample of sclerites of *X. concinnus* and *L. pseudominutus* was submitted for dating together with a further sample of the moss beetle *Simplocaria metallica* (Sturm). Unfortunately neither was sufficient to obtain a date.

3.5.3 Garðar

Two further samples were provided for dating from the manured fields adjacent to the Bishop's farm and the cathedral at Garðar in the Western Settlement. These were from the uppermost profile, close to the farm (Column A in Panagiotakopulu et al., 2012), and are likely to reflect the latest manuring events at the farm. Again, paired samples of a synanthropic species, here the spider beetle *Tipnus unicolor* Pill. & Mitt., which is a generalised detrital feeder, sometimes found in quantity in human faeces (e.g. Warsop and Skidmore, 1998), and a species drawn from the natural fauna, *S. metallica* were employed; neither yielded enough carbon for an AMS date.

4. Conclusions

Although the results obtained early in the project were promising (Table 3), others obtained later from securely dated contexts showed variation (Table 5, and several dates in Table 7, Fig. 2 and 3). The first batch of dates, Lateglacial Armthorpe and GUS dates from samples 3513 and 2790, were carried out before the development of the pre-treatment methods outlined by Tripp et al. (2004), and whilst the first is probably too young in relation to similar better dated faunal assemblages, the dates on insect chitin from GUS are too old by several hundred years and there is no chance that this reflects contamination by insects much older than the context. At GUS, setting aside the additional problems created by the marine reservoir effect on the dates from the contents of the soapstone pot, parallel dates on wood and a dung pellet appear correct and the problem is restricted to the chitin dates.

This was solved with the use of the new pre-treatment method (IA*) for the second batch of dates and by storage in distilled water, preferably slightly acidified to preclude mould growth. Although using the new method, the one date on material from the Roman well at Empingham is at least two hundred years too old, the dates from the Lynch Farm well, with the exception of one, appear to be within the timeframe from the archaeology.

Unfortunately it was not possible to track the sources of the various alcohols used in sample processing and storage, but the fact that dates are consistently older suggests that the sources for both GUS and Empingham were petrochemical rather than wood. One date from Lynch Farm remains problematic and could reflect either wood alcohol or taphonomic problems, although the possibility has also to be entertained that the water soluble gum tragacanth, used in mounting, had reacted with the chitin. There is evidence for cross-contamination of biological tissue after storage in ethanol. Barrow et al. (2008), for example, tested the effect on turtle remains and tissues of storage in a range of preservatives, including ethanol. They found mixed effects on $\delta^{13}C$ values, sometimes there were significant enrichments or depletions, other times not. For ethanol stronger than 70% there were more significant effects identified on stable isotope values. Kaehler and Pakhomov (2001) tested ethanol storage on fish tissues and discovered that this significantly increased the $\delta^{13}C$ values of the material. During their study of deep sea corals stored in ethanol for a year, on the other hand, Stzrepek et al. (2014) found no effect of ethanol on coral protein. These studies focus on predominantly short-term storage. Our results show that long term storage in ethanol may affect radiocarbon dates, and probably $\delta^{13}C$ values as well, although the differences between the values of ethanol and insect chitin are not significantly large enough to make these effects straightforward to identify.

The dates from desiccated and charred remains, where no solvents were used for purification or storage, are consistent with the archaeological or other dates from the same contexts. The anomalous date from the Turin mask insects reflects the posthumous penetration of bitumen into the insects which had fed on the fresh corpse during the embalming process. This explanation of contamination by bitumen also explains the problem with the dates from the human bone of Manchester Mummy 1770. The new date on desiccated insects preserved within the bandages of 1770 is more probably the correct one, providing with the bandage dates, a late Ptolemaic date, which does not require the special explanation of a later Roman rewrapping of the body.

In summary the results from the dating programme are:

- The new pre-treatment methodology has been successful for desiccated and charred remains and where no chemicals have been employed and for material stored in acidified water immediately after paraffin flotation and sorting in ethanol.
- Storage medium is critical and further research is needed to understand how different chemicals, principally alcohols, and long term storage in them affect chitin.
- Substances such as bitumen have an apparent effect on the chitin dates.
- Insect diets do not seem to play an important role for dating, although site and context taphonomy, including carbon reservoir effects, should be taken into account during interpretation of results.

We expect that by applying the more rigorous Method B (IB*, deacetylation of chitin), we should obtain more accurate determinations for all samples, but larger amounts of insect material are required and it is this which has precluded its wider use thus far.

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Table 6 Dates of insects from two well dated Roman wells from Empingham, Rutland and Lynch farm in Peterborough.

Table 7 AMS ^{14}C dates from organic materials, including insect remains from Gården under Sandet (GUS) in the Western Settlement, Greenland and unsuccessful attempt for dating chitin from Garðar in the Eastern Settlement.

Context	Insects dated	Diet	Processing/storage details
Armthorpe	*Plateumaris sericea* (L.)	phytophagous on littoral aquatic vegetation	paraffin flotation, storage in ethanol
Manchester Mummy 1770	*Necrobia rufipes* (Deg.)	protein feeder, found during unwrapping of the mummy, under the right leg	no chemicals used
Manchester Mummy 1770	*Chrysomya albiceps* (Weide.)	protein feeder, found during unwrapping of the mummy, under the right leg	no chemicals used
Turin Museum Ptolemaic mask	*Dermestes frischii* Kugelann	protein feeder, incorporated in resinous substance on Ptolemaic mummy's cartonnage mask	no chemicals used
Empingham Roman well	Carabidae indet.	Both invertebrate predator and graminivorous taxa	paraffin flotation, storage in ethanol
Lynch Farm Site 2 well I, B104	*Nebria brevicollis* (F.)	invertebrate predator, largely on fly larvae, Collembola and mites	paraffin flotation, storage in ethanol
Lynch Farm Site 2 well I, B104	*N. brevicollis*	invertebrate predator, largely on fly larvae, Collemboloa and mites	paraffin flotation, storage in ethanol
Lynch Farm Site 2 well I, B104	*Pterostichus niger* (Schall.)	invertebrate predator	paraffin flotation, storage in ethanol
Lynch Farm Site 2 well I, B104	*Amara aulica* (Panz.)	largely graminivorous/phytophagous but will also take invertebrate prey	paraffin flotation, storage in ethanol
GUS 3513 in 3369	*Heleomyza borealis* Bohe.	feeds largely on protein in carrion	paraffin flotation, storage in ethanol
GUS 2790/1	*H. borealis*	feeds largely on protein in carrion	paraffin flotation, storage in ethanol
GUS 2790/2	*Xylodromus concinnus* (Marsh.)	synanthropic species, probably a predator or mould feeder often associated with stored plant materials (hay)	paraffin flotation, storage in ethanol
GUS 2790/3	*Latridius pseudominutus* Strand	synanthropic species, a mould feeder associated with stored plant materials (hay)	paraffin flotation, storage in ethanol
GUS 2469	*Scatella* cf. *stagnalis* (Fallén)	feeds on surficial growth of green algae	paraffin flotation, storage in ethanol
GUS 2469	*Nysius ericae/groenlandicus* Zett.	graminivorous	paraffin flotation, storage in ethanol
GUS 3159	*L. pseudominutus* & *X. concinnus*	synanthropic species, a mould feeder and probable predator associated with stored plant materials (hay)	paraffin flotation, storage in ethanol
GUS 3159	*Simplocaria metallica* (Sturm)	phytophagous, observed feeding on mosses and lichens in Greenland	paraffin flotation, storage in ethanol
Garðar A 35-40cm	*Tipnus unicolor* (Pill. & Mitt.)	a strongly synanthropic species, associated with moderately dry, decaying animal materials	paraffin flotation, storage in ethanol
Garðar A 35-40cm	*S. metallica*	phytophagous, observed feeding on mosses and lichens in Greenland	no chemicals used

Sample	Material	Lab Code	Radiocarbon Age BP	Calibrated age (95.4% probability)	$\delta^{13}C$ (‰)
Armthorpe SE 637058	Bulk peat	N-810	11110 ± 200	11411-10739	_
Armthorpe SE 637058	*Carex* sp. nutlets	OxA-10897	11150 ± 180	11382-10761	26.1
Armthorpe SE 637058	*Plateumaris ericea* (L.)	OxA-10898	11330 ± 80	11383-11105	25.4

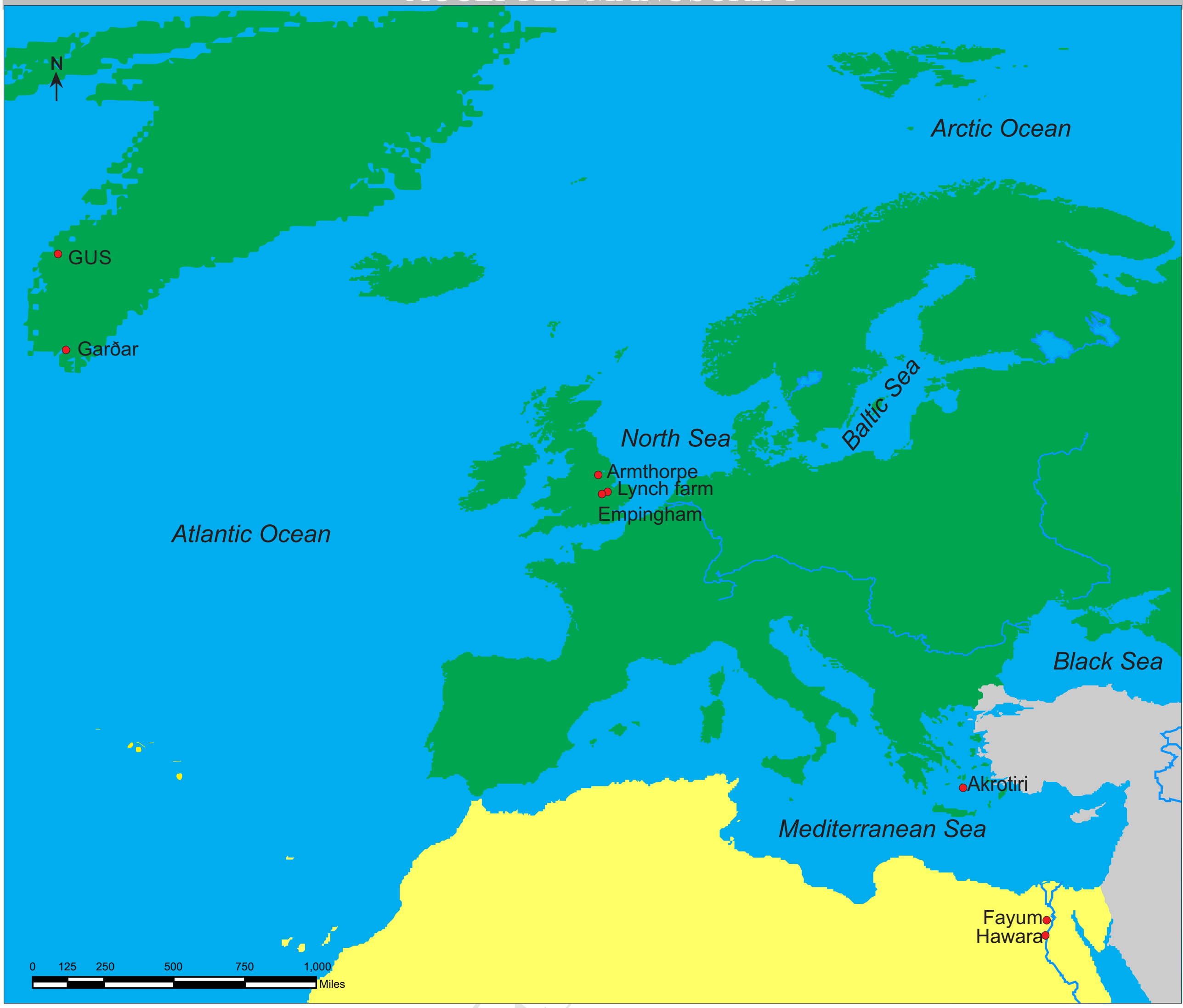
Sample	Material	Lab Code	Radiocarbon Age BP	Calibrated age (95.4% probability)	$\delta^{13}C$ (‰)
Akrotiri, West House, Room 5, Pithos 1	*Lathyrus clymenum* L.	OxA - 1548	3335 ±60	1756 BC -1459 BC	-26.0
Akrotiri, West House, Room 5, Pithos 1	*L. clymenum*	OxA - 1549	3460 ±80	2012 BC -1537 BC	-26.0
Akrotiri, West House, Room 5, Pithos 1	*Lathyrus cicera/sativus* L.	OxA -1550	3395 ±65	1880 BC -1529 BC	-26.0
Akrotiri, West House, Room 5, Pithos 1	*Bruchus rufipes* L.	OxA-25176	3368 ±29	1744 BC -1538 BC	-23.1

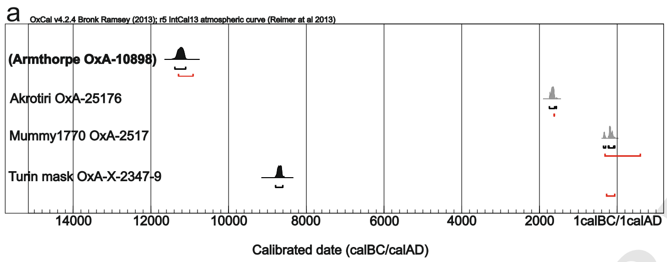
Sample	Material	Lab Code	Radiocarbon Age BP	Calibrated age (95.4% probability)	$\delta^{13}C$ (‰)
Manchester Mummy 1770	*Necrobia rufipes* (Deg.)	OxA-2517	2142 ± 26	352 BC - 62 BC	-21.85
Manchester Mummy 1770	*Chrysomya albiceps* (Weide.)	P-29455	_	_	_
Manchester Mummy 1770/469	Right scapula	Hodge and Newton (1979)	2780 ± 180	1426 BC - 510 BC	_
Manchester Mummy 1770	Left scapula	Hodge and Newton (1979)	2826 ± 173	1493 BC - 546 BC	_
Manchester Mummy 1770	Outer bandage	Hodge and Newton (1979)	1594 ± 126	AD 140 - AD 659	_
Manchester Mummy 1770	Part 4 bandage	Hodge and Newton (1979)	1713 ± 135	AD 25 - AD 605	_
Manchester Mummy 1770/169	Left humerus	BM-1602	2080 ±160	511 BC - 259 AD	-24.2
Manchester Mummy 1770	Skin tissue, left humerus	LJ-4915	2290 ± 40	408 BC- 208 BC	-25.37
Manchester Mummy 1770	Linen wrapping bandages over right side, arm, and chest	LJ-4995	2920 ± 60	1282 BC - 932 BC	-25.7
Manchester Mummy 1770	Chest linen bandages from beneath cantonnage chest cover	LJ-4996	2130 ± 60	362 BC - 3 BC	-26.2
Manchester Mummy 1770	Linen 16th layer from top over the legs /446	OxA-11650	2151 ±37	358 BC - 58 BC	-23.6
Manchester Mummy 1770	Linen wrapped around cartonnage mask /101	OxA-17824	1797 ±25	133 BC - 323 AD	-24.1
Turin Museum Ptolemaic mask	*Dermestes frischii* Kugelann	OxA-X-2347-9	9409 ± 38	8791 BC - 8606 BC	-24.03

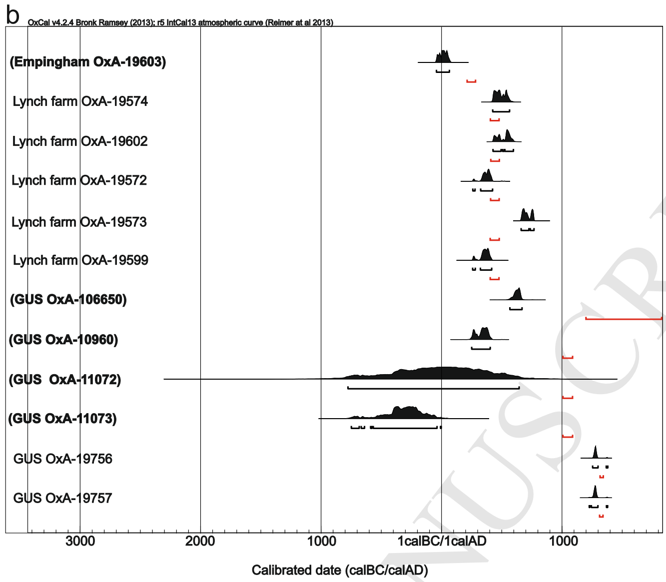
Sample	Material	Lab Code	Radiocarbon Age BP	Calibrated age (95.4% probability)	$\delta^{13}C$ (‰)
Empingham well	Carabidae indet.	OxA-19603	1986 ± 26	43 BC - AD 66	-24.7
Lynch Farm, Site 2, well I, B104	*Nebria brevicollis* (L.)	OxA-19574	1551 ± 25	AD 426 - AD 566	
Lynch Farm, Site 2, well I, B104	*N. brevicollis*	OxA-19602	1530 ± 24	AD 428 - AD 598	-25.7 -25.4
Lynch Farm, Site 2, well I, B104	*Pterostichus niger* (Schall.)	OxA-19572	1670 ± 27	AD 261 - AD 425	-25.3
Lynch Farm, Site 2, well I, B104	*Amara aulica* (Panz.)	OxA-19573	1300 ± 25	AD 662 -AD 769	-25.9
Lynch Farm, Site 2, well I, B104	Carabidae indet.	OxA-19599	1683 ± 26	AD 259 - AD 417	-25.2

Sample	Material	Lab Code	Radiocarbon Age BP	Calibrated age (95.4% probability)	$\delta^{13}C$ (‰)
GUS 3513 in 3369	Charred seaweed	OxA-10531	1354 ± 38	AD 614- AD 766	-14.3
GUS 3513 in 3369	*Heleomyza borealis* Bohe.	OxA-10665	1413 ± 39	AD 568 - AD 669	-22.7
GUS 2790/4	Twigs	OxA-10768	823 ± 40	AD 1058 - AD 1276	-26.2
GUS 2790/7	Ovicaprid dung pellet	OxA-11074	1005 ± 45	AD 904 - AD1147	-26.8
GUS 2790/1	*H. borealis*	OxA-10960	1703 ± 33	AD 251- AD 405	-21.5
GUS 2790/2	*Xylodromus concinnus* (Marsh.)	OxA-11072	1960 ± 320	777 BC- AD 645	-26.8
GUS 2790/3	*Latridius pseudominutus* Strand	OxA-11073	2250 ± 110	749 BC -3 BC	-26.8
GUS 2469	*Scatella* cf. *stagnalis* (Fallén)	OxA-19756	719 ± 27	AD 1255- AD 1381	-23.58
GUS 2469	*Nysius ericae/ groenlandicus* Zett.	OxA-19757	725 ± 28	AD 1227- AD 1380	-22.17
GUS 3159	*L. pseudominutus* & *X. concinnus*	P-22477	_	_	_
GUS 3159	*Simplocaria metallica* (Sturm)	P-22457	_	_	_
Garðar A (35-40cm)	*Tipnus unicolor* (Pill. & Mitt.)	P-22480	_	_	_
Garðar A (35-40cm)	*S. metallica*	P-22479	_	_	_

Geographic Area	Site/Context	Sample	Date based on Quaternary/ Archaeological information
Armthorpe, UK	pro-glacial Lake Humber	sample from thin peat lens	11100+/- 200 cal BP
Akrotiri, Santorini, Greece	West House, Room 5, pithos 1	infested pulses	1627-1600 cal BC
Hawara, Egypt	Mummy 1770 (Manchester Museum)	sample from upper right leg	Graeco-Roman (332 BC - AD 641)
Fayum, Egypt	Mummy cartonnage mask (Turin Museum)	insects in bitumen	Ptolemaic (305 BC - 30 BC)
Empingham, UK	Roman well	sample from the bottom of the well	AD 201 - AD 300
Peterborough, UK	Lynch farm, Well 1	B104	AD 400 - AD 500
Nuuk, Greenland	Gården under Sandet (GUS)	S3153	>AD 1200
Nuuk, Greenland	Gården under Sandet (GUS)	S2790, S3159	Phase 1-2 (AD 1000 - AD 1100)
Nuuk, Greenland	Gården under Sandet (GUS)	S2469	Phase 8 (AD 1300 - AD 1350)
Igaliku, Greenland	Garðar	column A, 35-40cm	AD 1100 - AD 1300


N
Arctic Ocean
GUS
Garðar
North Sea
Baltic Sea
Armthorpe
Lynch farm
Empingham
Atlantic Ocean
Black Sea
Akrotiri
Mediterranean Sea
Fayum
Hawara
0 125 250 500 750 1,000
Miles


a
OxCal v4.2.4 Bronk Ramsey (2013); r5 IntCal13 atmospheric curve (Reimer et al 2013)
(Armthorpe OxA-10898)
Akrotiri OxA-25176
Mummy1770 OxA-2517
Turin mask OxA-X-2347-9
14000
12000
10000
8000
6000
4000
2000
1calBC/1calAD
Calibrated date (calBC/calAD)


b
OxCal v4.2.4 Bronk Ramsey (2013); r5 IntCal13 atmospheric curve (Reimer at al 2013)
(Empingham OxA-19603)
Lynch farm OxA-19574
Lynch farm OxA-19602
Lynch farm OxA-19572
Lynch farm OxA-19573
Lynch farm OxA-19599
(GUS OxA-106650)
(GUS OxA-10960)
(GUS OxA-11072)
(GUS OxA-11073)
GUS OxA-19756
GUS OxA-19757
3000
2000
1000
1calBC/1calAD
1000
Calibrated date (calBC/calAD)

Highlights

- The pre-treatment methodology shows satisfactory results on desiccated and carbonised material
- Long term exposure in chemical solvents, primarily alcohols, may result in problematic dates
- Insect diets do not have an effect on the quality of the dates
- The taphonomy needs to be taken into account even when interpreting apparently secure dates